

Determination of Platinum, Palladium, and Lead in Biological Samples by Atomic Absorption Spectrophotometry

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A flameless atomic absorption method for the coextraction of platinum and palladium from biological and environmental samples by high molecular weight amine (HMWA) is given. Also, methods for lead determination in biological samples by use of extraction-flameless analysis and direct aspiration-flame analysis are reported. A study of lead contamination of Vacutainer tubes is given.

Introduction

Monitoring our environment for heavy metal pollution requires reliable and accurate analytical methods. Methods must also be developed for those metals which are not at the present sources of pollution but could become so through changes in technology and increased industrial usage.

The use of catalytic converters on U.S. automobiles has projected platinum and palladium into the role of possible environmental pollutants. It is necessary to establish baseline levels of these metals in the environment, prior to the widespread use of these catalytic converters, so that future monitoring of these metals in the environment can be evaluated.

A review of the literature, prior to this year, revealed almost no values for Pt or Pd in biological samples. Where values have been reported, the method of analysis has not been specific for Pt or Pd, and only an estimation of concentrations was made (1).

Due to the increased interest in Pt and Pd caused by the catalytic converters, more papers are becoming available on analysis of these metals in

biological tissues and fluids. A recent paper of Yoakum, Stewart, and Sterrett (2) cited an emission spectrochemical method to determine Pt, Pb, and Mn in rat tissues.

LeRoy (3) and Miller and Doerger (4) stated that to determine Pt in 1 g of wet tissues by the graphite analyzer, there would have to be a minimum concentration of approximately 0.2 ppm for reproducible results.

The analysis of Pt and Pd by use of the graphite analyzer and atomic absorption spectrophotometry (AAS) has been reported on aqueous solutions both directly and by extraction (5-9).

This paper reports on an atomic absorption spectrophotometric method for the determination of Pt and Pd in biological samples involving use of a high molecular weight amine (HMWA) extraction procedure. Methods for the determination of Pb in biological samples will also be reported.

Trace metal studies are always plagued by contamination problems. Evidence that "low-lead" Vacutainer (Becton-Dickinson Co., Rutherford, N. J.) blood collection tubes (10 ml) were contaminated with more Pb than the manufacturer certified prompted a "leach" study of the Vacutainers used in our laboratory. Platinum and palladium analyses were included in this study.

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Experimental

Instrumentation

All analyses were performed on a Perkin-Elmer Model 306 atomic absorption spectrophotometer modified with Perkin-Elmer optical modification kit (No. 040-0286) to reduce the "light scattering" effect caused by the graphite furnace. A deuterium arc background corrector was routinely used for all metal determinations. Absorption peaks were recorded with a Perkin-Elmer 056 recorder. Flameless analyses were performed with a Perkin-Elmer HGA-2000 graphite furnace. Some analyses were by aspiration of the sample into an air/acetylene flame by using a three-slot burner head.

Calculation of least-square fit of regression curves (method of additions) to determine analyte concentrations was performed on a Hewlett-Packard programmable calculator, Model 9810A.

Reagents

All reagents were analytical grade unless otherwise specified. Atomic absorption standard solutions (1000 ppm) for Pt and Pd were the H_2PtCl_6 and $PdCl_2$, respectively, from Ventron Corp., Alfa Products, Danvers, Md. Atomic absorption standard for Pb (1000 ppm) was from Fisher Scientific Co., Pittsburgh, Pa.

Analytical Considerations

Several important criteria were considered in developing methods for Pt, Pd and Pb analyses of biological materials: (1) the expected low concentration of Pt and Pd would require some preconcentration procedure; (2) the method for each metal had to be simple, fast and accurate, as a large number of samples had to be analyzed in a relatively short period of time; (3) preferably, the preconcentration step (extraction) should remove Pt, Pd and Pb, to reduce the time needed for sample preparation; (4) the quantity of sample available for analysis would be limited and therefore place restrictions upon the limit of detection of Pt and Pd; (5) finally, the time available for methodology development of Pt and Pd procedures was limited.

Platinum and Palladium Procedure

Considering the above restrictions, the main emphasis of our work was to find a suitable extraction procedure which would quantitatively remove both Pt and Pd from the ashed or digested sample

with a single extraction. The extraction procedures of Khattak and Magee (10,11) which utilize a "liquid anion exchanger" (12) in the form of a tertiary high molecular weight amine appeared to be the best choice.

This extraction procedure was modified to determine Pt and Pd by AAS. Blood is used to demonstrate the procedure, but it is adaptable to other sample matrices.

Blood: A 15-ml sample of whole blood was wet digested with HNO_3-HClO_4 (50:50) in a Vycor beaker under N_2 on a hot plate (150°C). The digest solution was evaporated to near dryness and reconstituted three times with 5 ml of concentrated HCl. Following the last reconstitution, the sample was removed from the hot plate and allowed to cool before rinsing into an extraction vessel with 3N HCl. A 1-ml portion of a 25% $SnCl_2$ solution in 3N HCl was added, followed by 1 ml of 0.02M tri-*n*-octylamine (in xylene). The vessel was shaken for 15–20 min and then centrifuged at 3000 rpm for 10 min. The octylamine layer was removed, and a second extract with 1 ml of xylene was carried out following the same procedure. The two extracts were combined in a glass vial and evaporated to dryness. The vials were sealed and the samples maintained in the dry state until ready to analyze, at which time they were reconstituted with 0.1 ml of xylene. A 5–10- μ l aliquot of this extract was injected into the HGA-2000 graphite furnace for Pt and Pd determinations.

Composites: Composite samples of urine containing up to 1 liter were evaporated to dryness using a rotary evaporator. The dry residue was then ashed in a muffle furnace at 500°C. Aqua regia was used to solubilize the ash, and this solution was then evaporated to near dryness and reconstituted three times with concentrated HCl. The final solution was diluted and extracted by the procedure given for blood.

Feces and hair composites containing 300 g and 11.5 g, respectively, were wet ashed in a Vycor beaker with HNO_3-HClO_4 (50:50). The digests were continued until a clear solution remained. The samples were then evaporated to near dryness and reconstituted several times with concentrated HCl. The solution was then diluted and extracted by the above procedure.

Lead Procedure

Blood: The procedure of Hwang, Ullucci, and Mokeler (13) with modifications from the method of Mitchell, Ryan, and Aldous (14) was used to determine the Pb concentration in whole blood samples.

An Eppendorf pipet was used to transfer a 1.0-ml aliquot of whole blood into a 5-ml centrifuge tube. A 1-ml portion of a hemolyzing-chelating solution consisting of a 5% Triton X-100 (octylphenoxypolyethoxyethanol, Rohm and Haas, Philadelphia, Pa.) solution containing 2% ammonium pyrrolidine dithiocarbamate (APDC) was added, and the sample was mixed vigorously for 5 min and allowed to stand 10–15 min to ensure complete hemolysis of the blood; this was followed by the addition of 1.0 ml of water-saturated MIBK. The sample was then shaken for 3 min and centrifuged for 5 min at 3000 rpm. The organic layer was then analyzed for Pb by injecting 10- μ l aliquots into the HGA-2000 graphite furnace.

Urine: The procedure used for urinary Pb determinations was based upon that of Kubasik and Volosin (15).

A 10-ml aliquot of acidified urine (1% acetic acid added as a preservative) was pipetted into a 25-ml centrifuge tube. NaOH was used to adjust the pH to 7.0, and 5 ml of tris(hydroxymethyl)-aminomethane buffer (pH 7.0) was added. A 1-ml portion of a 1% APDC solution was added along with 1.0 ml of water-saturated MIBK. The sample was mixed for 10 min and then centrifuged for 10 min at 3000 rpm; the organic layer was then removed for Pb analysis by injecting aliquots into the HGA-2000 graphite furnace.

Hair: Hair samples were washed by a modification of the method of Hammer et al. (16).

All of the hair sample collected (usually 1–4 g) was cut into approximately 1-cm lengths with stainless steel scissors. The hair was placed in a 250-ml Erlenmeyer flask, and a sufficient amount of 0.12% sodium lauryl sulfate solution was added to cover the hair completely. The flask was then placed on a mechanical shaker for 1 hr; then the solution was decanted off, and the hair was rinsed with deionized water until no trace of the surfactant remained. The hair was then washed twice with isopropyl alcohol and rinsed three times with deionized water. The washed hair was placed in a polyethylene beaker, covered with thin paper to keep contamination out, and placed in an oven (60°C) until dry.

A 1-g sample of the washed and dried hair was weighed into a Vycor beaker and digested on a hot plate with HNO₃—HClO₄ (50:50). The digest was evaporated until dense fumes of perchlorate were given off and was then allowed to cool. The digest was filtered with a glass-fiber filter, 0.1N HNO₃ being used as rinse. The filtrate was collected in a 10-ml volumetric flask and make to volume with 0.1N HNO₃. This sample was analyzed for Pb by aspirating into an air/acetylene flame.

Feces: A 1.5-g aliquot of homogenized feces was digested with concentrated HNO₃ in a Teflon bomb designed by Rantala and Loring (17). The bomb was placed on a hot plate at 150°C for 1 hr, removed, and allowed to cool to room temperature before attempting to open. The digest was quantitatively rinsed through a glass fiber filter, and the filtrate was collected in a 10-ml volumetric flask. Deionized water was used to make the volume to 10 ml. This solution was analyzed for Pb by injecting 10- μ l aliquots into the HGA-2000 graphite furnace.

Instrument Parameters

Table 1 lists the instrument parameters used for analysis of Pb by the flame method.

Table 1. Instrument parameters for lead determination by air/acetylene flame.

Parameter	
Spectrophotometer	
Wavelength, nm	283.3
Source, mA	9
Slit	No. 4 (1.0 mm)
Damping	No. 1
D ₂ arc	ON
Recorder, mm/min	20
Scale expand	Auto Conc. with 5 ppm Pb at full scale; also 2.5 ppm full scale
Flame	
Air, psi	30 (No. 60)
Acetylene, psi	8 (No. 40)
Aspiration rate, ml/min	2.5

Table 2 gives the parameters for Pt, Pd, and Pb analyses on the HGA-2000 graphite furnace.

Table 2. Instrument parameters for the HGA-2000 determination of Pt, Pd, and Pb.

Parameter	Lead	Platinum	Palladium
Spectrophotometer			
Wavelength, nm	283.3	265.9	247.6
Source, mA	9	18	23
Slit	No. 4 (1.0 mm)	No. 4 (1.0 mm)	No. 3 (0.3 mm)
Damping	No. 1	No. 1	No. 1
D ₂ arc	ON	ON	ON
Recorder, mm/min	10	10	10
Scale expand	1 time	3 times	10 times
Graphite furnace			
Dry, sec/°C	25/ 100	15/ 200	30/ 150
Ash, sec/°C	25/ 500	30/1500	30/ 500
Atomize, sec/°C	7/2100	15/2700	15/2700
Tube	Grooved	Regular	Regular
Gas	N ₂ at 20 psi, No. 4 (flow-meter), auto interrupt	N ₂ at 20 psi, No. 4 (flow-meter), manual interrupt	N ₂ at 20 psi, No. 4 (flow-meter), manual interrupt

Standard Solutions

Platinum and Palladium: Quantitation of blood samples for Pt and Pd was accomplished by spiking 15-ml aliquots of whole blood prior to digestion with 50 μ l and 100 μ l of a combined aqueous standard containing 1.2 μ g/ml Pt and 0.2 μ g/ml Pd. These spiked samples represented blood concentrations of Pt of 0.004 and 0.008 μ g/ml and of Pd of 0.00067 and 0.0013 μ g/ml.

Composite samples were spiked according to Table 3 by using an aqueous standard containing 2 ppm Pt and 1 ppm Pd.

These spiked samples (blood and composites) were used to quantitate the Pt and Pd concentrations of the unknown samples.

Lead: Pb spikes were added to each type of sample matrix according to Table 4.

Interferences

Concentrations of 500 to 1000 ppm of Fe^{3+} , Mn^{2+} , Cu^{2+} , Pb^{2+} , Cd^{2+} , and Zn^{2+} were added to

Table 3. Pt and Pd spike levels of composite samples.

Sample	Quantity of sample analyzed	Spike solution used, μ l ^a	Concentration, ppm	
			Pt	Pd
Urine	1000 ml	500	0.0010	0.0005
		100	0.0002	0.0001
Feces	300 g	500	0.0033	0.0017
		100	0.0007	0.0003
Hair	11.5 g	50	0.0087	0.0043
		100	0.0174	0.0087
		200	0.0348	0.0174

^aSpike solution: 2 μ g/ml Pt and 1 μ g/ml Pd.

Table 4. Pb spike levels used to calculate analytical curves.

Sample	Quantity of sample analyzed	Spike solution, μ g/ml	Volume of spike solution used, μ l	Concentration of Pb in samples
Blood	1 ml	10	0	Natural concn
			5	5 μ g/100 ml
			10	10 μ g/100 ml
			20	20 μ g/100 ml
			30	30 μ g/100 ml
Urine	10 ml	10	0	Natural concn
			10	10 μ g/l
			20	20 μ g/l
			40	40 μ g/l
Hair	1 g	1000	0	Natural concn
			5	5 μ g/g
			10	10 μ g/g
Feces	1.5 g	100	25	25 μ g/g
			0	Natural concn
			5	0.33 μ g/g
			15	1.00 μ g/g
			30	2.00 μ g/g
			60	4.00 μ g/g

10 ml of a 3N HCl solution containing 0.6 μ g Pt and 0.1 μ g Pd. The solution was extracted by the procedure outlined earlier. None of these cations produced a change in the Pt peak heights greater than the inherent deviation of the analytical procedure. There is a significant enhancement of the Pd signal by Fe^{3+} , Mn^{2+} , and Cu^{2+} . An increasing loss of Pd has been observed as the HCl concentration of the sample increases above 3N. The same effect is noticed for Pt but does not start until the acid concentration is greater than 6N.

A serious loss of Pt spikes has been observed if the sample is digested with HNO_3 without evaporation and reconstitution several times with concentrated HCl. This is thought to be due to the formation of nitrosoplatinic complexes which must be destroyed before attempting to form the stannous-chloro complexes of Pt prior to extracting.

Analytical Data

Limit of Detection: The limits of detection for Pt, Pd, and Pb in the different sample matrices are given in Table 5. The limit of detection represents the concentration of analyte which will give a signal-to-noise ratio of 2.

Precision: Ten determinations made on a spiked blood sample gave a Pt concentration of 0.012 μ g/ml with a standard deviation of 0.0004 μ g/ml (C.V. = 3.3%) and a Pd concentration of 0.003 μ g/ml with a standard deviation of 0.00012 μ g/ml (C.V. = 4.1%).

Lead precision data are listed in Table 6.

Accuracy: At the present time, interlaboratory studies are being organized to ascertain the accuracy of these methods by techniques involving

Table 5. Limit of detection.

Sample	Detection limit, ppm		
	Pt	Pd	Pb
Blood	0.0014 ^a	0.0004 ^a	0.021
Urine	2×10^{-5b}	7×10^{-6b}	0.0047
Hair	0.0019 ^b	6×10^{-4b}	2.67
Feces	7×10^{-5b}	2×10^{-5b}	0.391

^aUsing 15 ml of blood.

^bComposite samples: calculated limit of detection.

Table 6. Precision for Pb analysis.

Sample	n	Pb concentration, ppm	S. D., ppm	C. V., %
Blood	10	0.178	0.012	6.74
Urine	12	0.01854	0.00292	15.75
Hair	12	26.21	1.799	6.86
Feces	8	0.265	0.184	69.43

analytical methods other than atomic absorption spectrophotometry.

Recovery of added spikes range from 97 to 101% for Pt and Pd in blood at 0.012 ppm and 0.003 ppm levels, respectively.

Lead recovery of spiked blood (0.2 ppm) range from 96 to 106%, while the recovery range for urine (0.002 ppm) is 89 to 97%. Recoveries of spiked lead in hair (10 ppm) and feces (1 ppm) range from 95 to 103%.

Vacutainer Study

Selection of Vacutainers: Each Vacutainer in a case lot (1000 Vacutainers) was numbered in a systematic manner. Then a series of random numbers were generated (Hewlett-Packard Model 9810A calculator) and used to select forty (40) Vacutainers for analyses.

Protocol: The "leach" solutions used for Pb analysis were whole blood, deionized water and dilute acid (0.1N HCl). The indigenous concentration of Pb in each of these solutions was analyzed.

Blood was the only leach solution used in the Pt and Pd analyses, and it was spiked at 0.2 ppm Pt and Pd concentrations.

Whole blood and deionized water were added to the Vacutainer by the route commonly used, i.e., with a needle using the vacuum in the tube. The dilute acid leach was added by removing the rubber stopper. Of the Vacutainers, 25 were used for whole blood, and the remainder were divided between the deionized water and dilute acid leaches.

All the Vacutainers were filled at the same time. Then at 1-, 3-, 6-, 24-, and 30-hr intervals, the blood tubes were analyzed for Pb. Vacutainers with deionized water were analyzed for Pb at 6-, 24-, and 30-hr intervals, and those with dilute acid were analyzed at 6 and 30 hr. Only one analysis was made for Pt and Pd, and that was at 40 hr. All the Vacutainers were maintained at room temperature throughout the study.

At each of the specified times, blood lead analysis was performed on five of the blood-containing Vacutainers and on three control blood aliquots (i.e., blood not exposed to a Vacutainer).

Three of the Vacutainers containing the other leach solutions were analyzed at the specified time intervals along with two control aliquots (i.e., deionized water and dilute acid leach solution not exposed to a Vacutainer).

Analysis: Lead analysis was performed by the method outlined earlier for blood lead determinations. The dilute acid leach samples were neutralized (pH 7.0) with NaOH prior to extraction.

Platinum and palladium analyses were performed on 5 ml of the blood leach solution by the extraction procedure given earlier.

Results and Discussion

Platinum and Palladium

An extraction system designed to handle a large number of samples on a routine basis must keep procedural steps to a minimum to avoid costly mistakes.

The high molecular weight amine (HMWA) extraction system described here is limited by the fact that the anionic exchange resin creates an excessive amount of "smoke" during the atomization step in the HGA-2000. This restricts the total amount of extract that can be analyzed and thereby limits the sensitivity of the method. Increasing the ashing temperature above 1500°C results in severe losses of both Pt and Pd. Increasing the ashing time has lengthened the time for each analysis without providing any increase in sensitivity.

Reducing the concentration of the anionic resin is a method of reducing the excessive "smoke", but Davidson and Jameson (18, 19) have shown that the concentration of other anions present will limit the extraction of the desired anions. Reducing the resin concentration may therefore cause incomplete extraction of the Pt and Pd species.

This excessive smoke upon atomization necessitates that the hollow cathode lamp and deuterium arc lamp be aligned very carefully, or incorrect absorption readings will result. Pd is affected more by incorrect alignment than is Pt.

Composite samples were another means of increasing the sensitivity for Pt and Pd. Experience in this laboratory has shown that wet ashing large quantities of solid material requires an excessive amount of time. Digestion of spiked composite hair and feces samples gave very erratic results. Recoveries were in the range 30–70% for Pt and Pd in feces and hair.

Spiked urine composite recoveries for Pd ranged from 0 to 60%, and for Pt the range was 0–100%. More work needs to be invested in this area to make it a reliable method for increasing the sensitivity of Pt and Pd determinations.

The limits of detections given in Table 5 for urine, hair, and feces are calculated on the basis of complete (>90%) recovery using the extraction procedure outlined. The limit of detection for blood is calculated on the basis that the absolute limits of detection of Pt and Pd, respectively, for our instrument are 1.07 and 0.33 ng.

Lead

The extraction procedure for blood Pb provides a simple and fast means of analyzing a large number of samples. Table 6 shows that the method has good precision.

Table 4 gives the quantity of spike solution added to 1 ml of blood to determine the analytical regression curve (Fig. 1) used to calculate the unknown blood samples. Attempts at spiking a large quantity of blood at different levels of Pb concentration and then using an aliquot for analysis have produced erratic results in our laboratory. The spike is added directly to the sample prior to extraction.

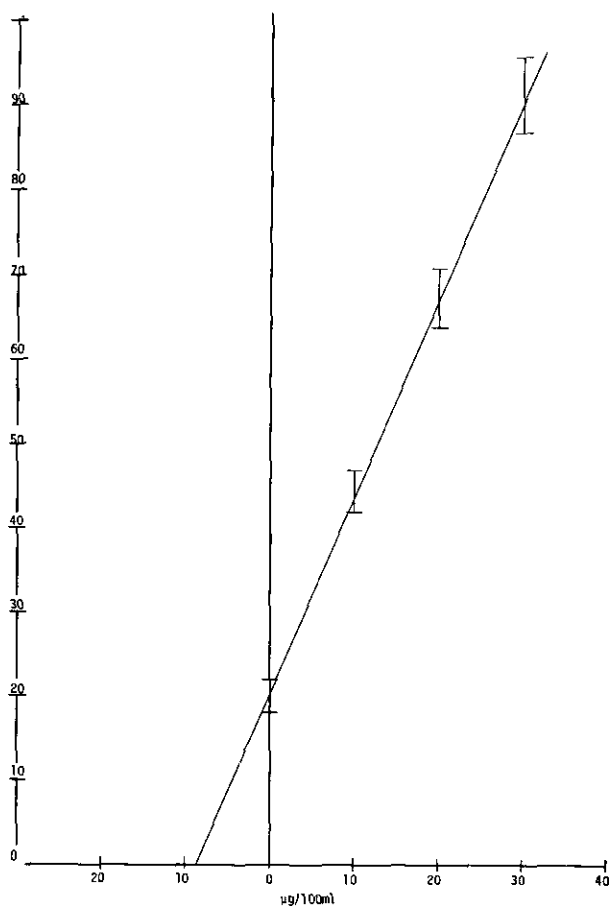


Figure 1. Regression analysis of blood lead standards. Lead concentration, $8.47 \pm 0.62 \mu\text{g}/100 \text{ ml}$; C. V. = 7.3%.

The limit of detection for Pb in blood given in Table 5 is restricted more by the reagent blank signal than by the background noise level of the instrument. Attempts to remove Pb from the reagents has been only partly successful.

Extraction of urine Pb by this procedure gives a rather high coefficient of variation (C.V.) (Table 6).

One reason for this is the low concentration of Pb normally found in urine. Another reason may be that the 1% acetic acid used in this laboratory to preserve the urine samples is not sufficient to maintain all the urine components in solution.

Like blood, the limit of detection of Pb in urine (Table 5) is a function of the Pb concentration of the reagent blank. High reagent blank absorption increases the limit of detection. Urine samples are spiked similarly to the method given for blood (Table 4) and for the same reason.

Hair analysis for Pb by the air/acetylene flame provides good precision (Table 6). The most common reason for deviations greater than 7% is improperly washed hair.

The limit of detection for Pb in hair by the flame method (Table 5) is limited by the instrument noise level rather than reagent blank interference. Spikes are added to the hair samples right after the digestion acid has been added.

Feces analysis for Pb varies (Table 6) because it is difficult to obtain a completely homogeneous sample. Even though the samples are homogenized prior to analysis, the relatively small sample analyzed (1.5 g) makes it difficult to improve upon the analytical variation.

Digestion of the feces in the Teflon bombs is a fast and easy method to analyze for Pb. The limit of detection is restricted by the Pb concentration in the reagent blank and the size of sample which may be used. Use of suprapure acids has decreased the reagent blank absorption but has not completely eliminated it.

Vacutainer Study

Calculations and Statistics: Blood aliquots spiked at four different concentrations were analyzed in duplicate. Regression analysis was performed with a programmable calculator on the absorption peak heights versus the Pb concentration. The results are illustrated by Figure 1. The blood-lead concentration was $8.47 \pm 0.62 \mu\text{g}/100 \text{ ml}$ (C.V. = 7.3%).

The results were calculated on the basis of the mean value for each sample. The test to show whether or not there is Pb contamination from the Vacutainers is a test for the difference between means of the independent groups using Student's *t* distribution.

Results: At the 0.05 level there is no significant difference between the control blood and the Vacutainer blood after 1 hr contact time (Table 7); however, there was a significant difference at the 3- and 30-hr intervals. At the 6- and 24-hr intervals, the difference between the controls and Vacutainer blood may or may not be significant.

Table 7. Results of Vacutainer study: comparison of controls and Vacutainer blood lead.

Time, hr.	Control blood, $\mu\text{g}/100\text{ ml}$		Vacutainer blood, $\mu\text{g}/100\text{ ml}$		Significant difference at the 0.05 level
	Mean	S.D.	Mean	S.D.	
1	8.47	0.28	9.65	1.01	No
3	8.47	0.41	10.25	0.85	Yes
6	8.47	1.43	9.76	1.58	?
24	8.47	0.66	9.51	1.22	?
30	8.47	1.20	9.75	0.73	Yes

Both the deionized water and dilute acid leaches showed significant differences between the controls and Vacutainers after 6 hr, but at 24 and 30 hr, there were no significant differences.

There was no indication of contamination of the Vacutainers with Pt or Pd. After 10 hr, there was a significant difference between the spiked controls and Vacutainers (Table 8), but this difference indicated a "loss" of the Pt and Pd spike to the container.

Table 8. Results of Vacutainer study: Pt and Pd.

	Platinum		Palladium	
	Control	Vacutainer	Control	Vacutainer
Peak height, mm	13	12	62	56
	8	7	54	56
	8	8	67	53
	11	5	69	
	5	11		
Mean	11	8.6	63	55
S. D.	3.1	2.9	6.7	1.7
t (0.05)		2.53		3.39
df		8		5
Significant		Yes		Yes
% Difference (means)		22		13

Conclusions

Platinum and Palladium

The extraction of Pt and Pd in a single extract by using a liquid anion exchanger (tri-*n*-octylamine) from an approximately 3N HCl solution provides a means of concentrating these elements so they will be within the detection limit of AAS by using the flameless graphite furnace. The procedure improves the limit of detection for Pt and Pd in biological samples and also provides a means of improving the sensitivity of AAS analysis for Pt and Pd.

The method is not restricted to biological samples but may be applied to environmental samples such as air, soils, and water.

Lead

The procedures for Pb analysis in blood, urine, and hair provide fast and reliable methods of determining Pb in these commonly used epidemiological monitors of environmental health.

Determination of Pb in fecal samples is considerably faster than by "open" digestion methods, but precision suffers because of the non-homogeneity of the sample.

Vacutainer Study

The lead contamination found in this particular case of B-D Vacutainer 10-ml tubes is approximately 0.13 $\mu\text{g}/\text{tube}$ which is essentially the 0.1 $\mu\text{g}/\text{tube}$ maximum level specified by the manufacturer's certification.

There was no evidence that the Vacutainers contained detectable amounts of either platinum or palladium. The data do indicate that after exposing spiked blood to the Vacutainers for 40 hr there is a 22% "loss" of the platinum spike and 13% "loss" of the palladium spike.

There should be no problem with contamination when these Vacutainers are used for epidemiological investigations.

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